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(71) Applicant
British Steel Corporation (United Kingdom),
9 Albert Embankment, London SE1 7SN

(72) Inventors
Gene Donald Spenceley
Raymond Jowitt

(74) Agent and/or Address for Service
Fry Heath & Co,
Seloduct House, Station Road, Redhill, Surrey

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(56) Documents cited
GB A 2130092 GB 1587723 EP A1 0047094
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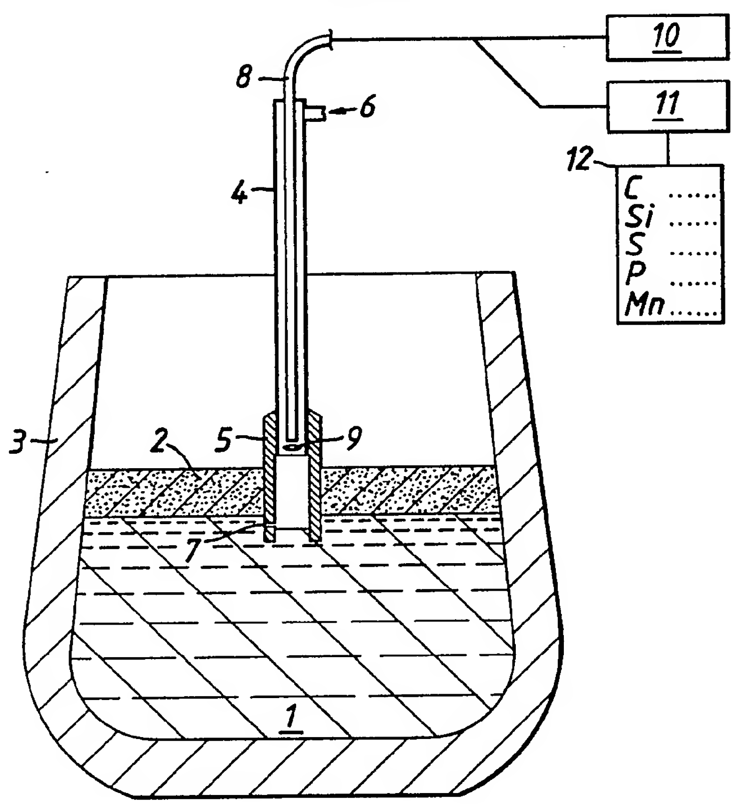
G1A

(54) Improvements in or relating to the analysis of materials

(57) The invention provides an arrangement for analysing a material comprising the steps of exciting a portion of the material by means of a pulsed beam from a laser 10, transmitting the radiation produced by the excitation of the material through a light guide 8 and analysing the radiation so transmitted in a spectrometer 11 including facility for analysis in the infra-red region of the spectrum.

FIG. 1.



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The drawing(s) originally filed was/were informal and the print here reproduced is taken from a later filed formal copy.

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FIG.1.

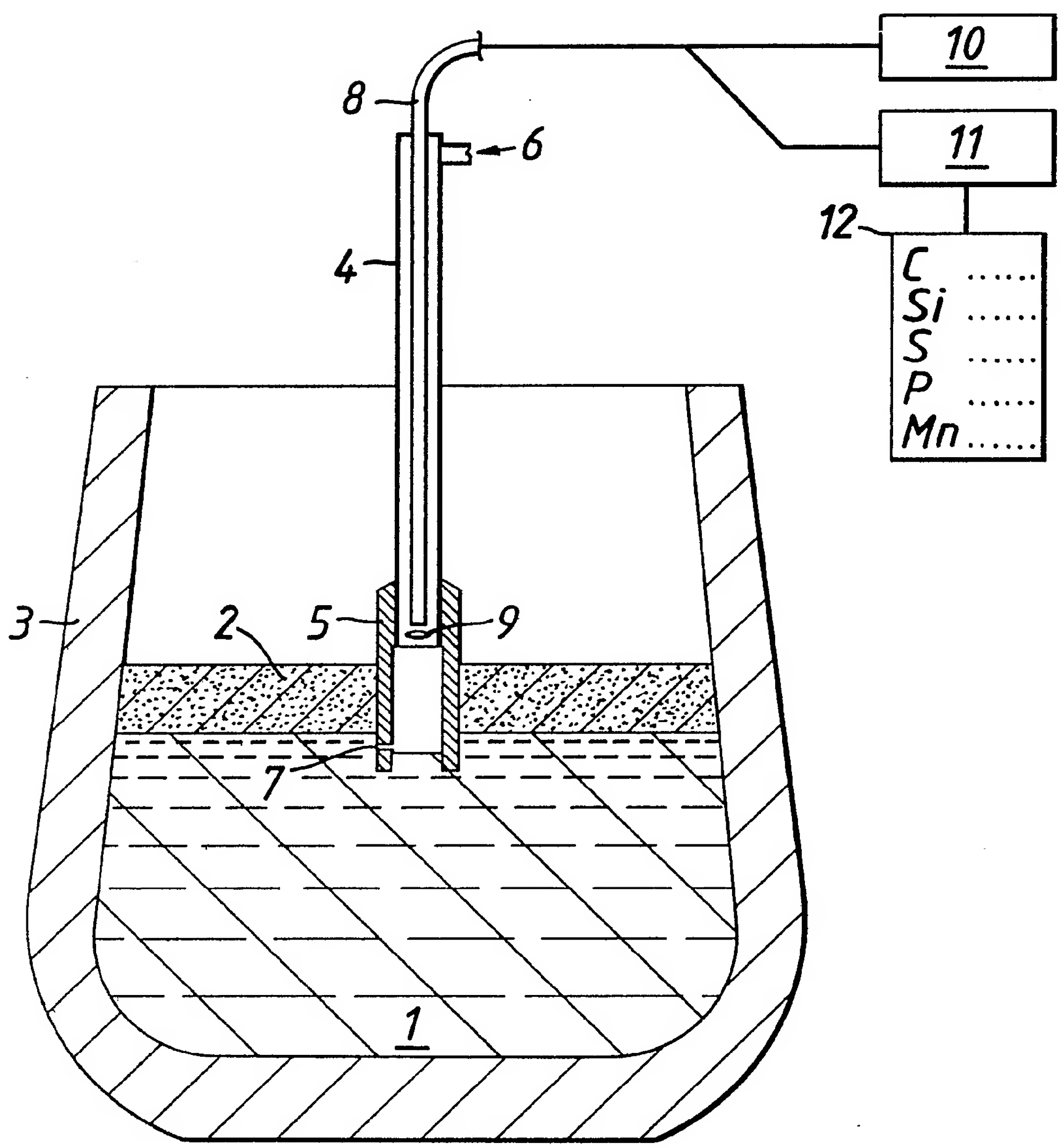


FIG. 2.

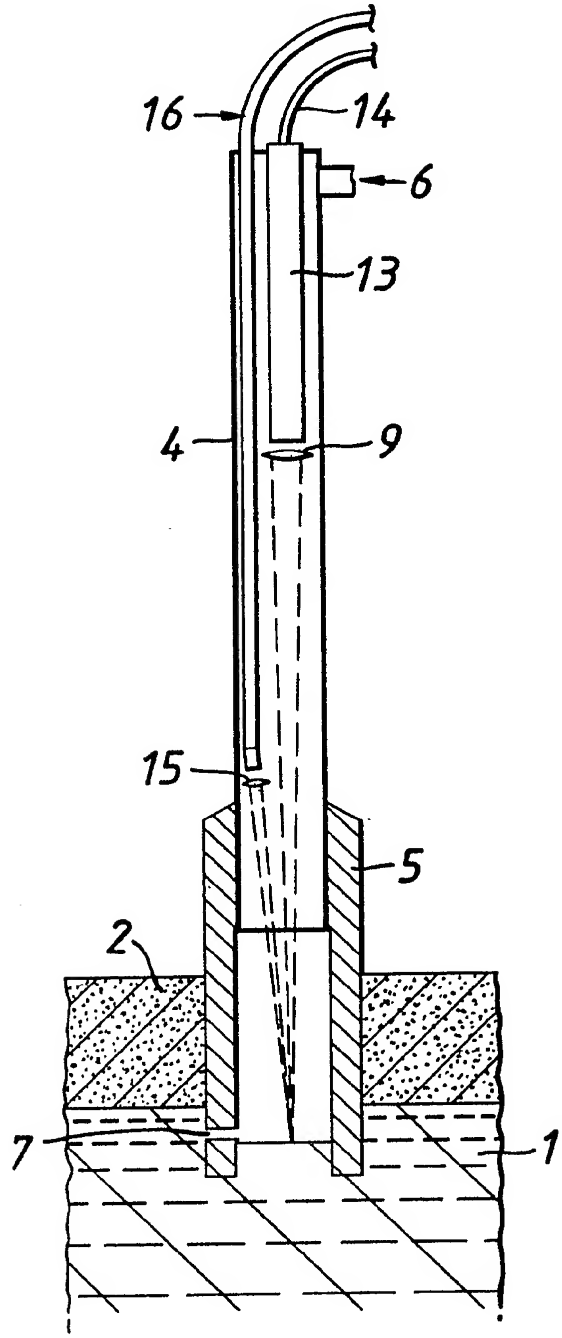
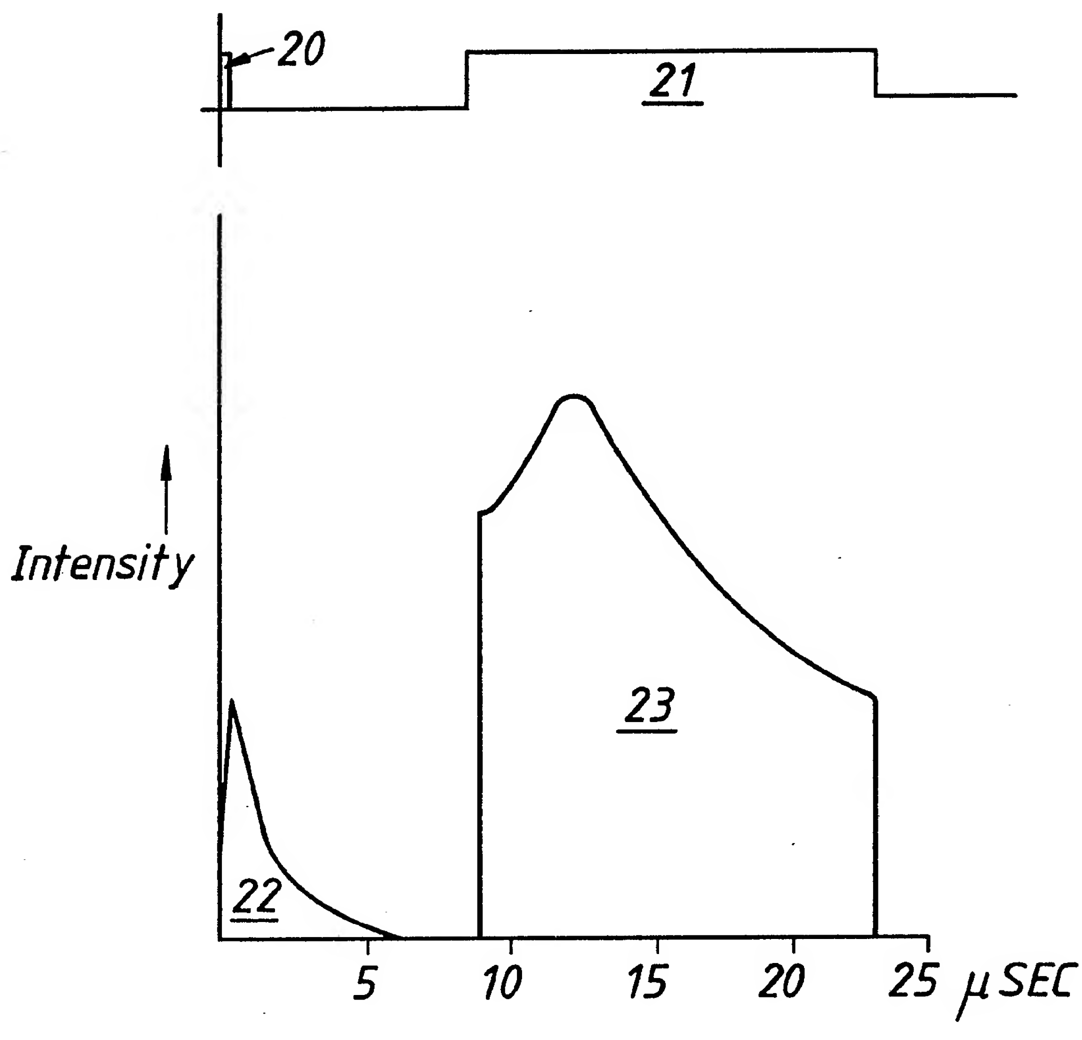


FIG. 3.



SPECIFICATION

Improvements in or relating to the Analysis of Materials

This invention relates to the analysis of materials.

According to one aspect of the invention there is provided a method of analysing a material comprising the steps of exciting a portion of the material by means of a pulsed laser beam, transmitting the radiation produced by the excitation of the material through a light guide and analysing the radiation so transmitted in a spectrometer including facility for analysis in the infra-red region of the spectrum.

According to another aspect of the present invention, there is provided apparatus for analysing a material comprising means for generating a pulsed laser beam for exciting a portion of the material, a light guide for transmission of the radiation produced by the excitation of the material to a spectrometer; the spectrometer incorporating analysing means working in the infra-red region of the spectrum.

The invention is especially, although not exclusively, applicable to the analysis of molten metals in a melting, refining or containing vessel; or solid material such as ferrous scrap for classification purposes; or of metal from a melt allowed to solidify in a liquid metal probe; or of slag material from a metal melt.

The use of a laser beam ensures a practical and suitable source of power for excitation purposes, and the pulsing beam enables the provision of sufficient power for excitation with the use of a reasonably sized generator. Thus, with liquid steel analysis, adequate excitation power has been provided with a 0.75 watt laser. Pulses of a few (e.g. 20) nanoseconds duration have been found appropriate.

The spectrometer may be gated to receive transmitted radiation a predetermined time after excitation of the material to improve the signal to noise ratio (as hereinafter explained).

The invention is based partly upon the realisation that light guide means can be used with great convenience and suitability for transmitting radiation. However, one serious difficulty with light guides is the transmission of, for example, carbon, sulphur and phosphorus spectral lines. Appropriate lines in the ultra-violet range are effectively absorbed by the glass or quartz from which light guides are manufactured. This problem is overcome in the invention by the provision of facility for analysis in the infra-red region of the spectrum, the analysis may be in the range 250nm to 1100nm which covers the infra-red range of approximately 700nm to 1100nm, and indeed the invention is also based upon the realisation that, contrary to expectation, the relatively weak infra-red spectral lines for such elements can provide quantitative analytical information via a light guide.

The Applicants have found that whereas, as suggested above, no material capable of use in a light guide has yet been identified having a sufficiently high transmission index in the <200nm region of the spectrum (where the carbon and sulphur and phosphorus spectral lines presently used for analysis are located) to permit determination of these elements with any certainty, the appropriate spectrum lines in the infra-red region (such as those around 900nm) of the spectrum can be transmitted through a glass or quartz light guide and enable the analysis of carbon, sulphur and phorphorus, provided the radiation from the metal excitation is sufficiently strong.

The spectrometric analysis apparatus may comprise a photodiode array detector or a photo-multiplier arrangement.

The laser is arranged to provide a pulsing beam, spectral measurements being made in a synchronised time gated sequence so as to receive excited radiation generated from the sample being tested but not general radiation. Additionally, or in the alternative, the laser may be subjected to variable tuning so as to provide optimum excitation for various spectral lines.

Where the apparatus is for use in the analysis of molten metal in a vessel, the light guide *per se* may, but need not, contact the molten metal. The apparatus may be arranged for continuous or semi-continuous sequential operation, in which case the light guide or light guides may be provided with thermal protection from the molten metal. Alternatively, the apparatus may be arranged for single individual readings at discrete intervals so that the light guide or guides need only be physically associated with the molten metal intermittently. In this case the guide or guides may be provided with disposable tips.

The light guide or guides may be introduced into the vessel through the top or through the side walls thereof.

The apparatus may incorporate temperature sensing means arranged to operate using radiation from the metal body transmitted through the light guide.

In order that the invention may be more readily understood, two embodiments thereof will now be described by way of example with reference to the accompanying drawings in which:—

Figure 1 is a diagrammatic representation of apparatus for the analysis of molten steel in a containing vessel;

Figure 2 illustrates an alternative arrangement of part of the apparatus of Fig. 1; and

Figure 3 is a graph showing time gating operation of the spectrometer.

It will be seen from Fig. 1 that steel 1 having a coating layer of slag 2 is contained

within vessel 3.

Projecting into the vessel 3 so as to penetrate to the surface of the steel below the level of the layer of slag 2 is a probe 4. The probe is protected at its end by means of a ceramic collar 5 suitably cooled and pressurised, to prevent damage by and entry of metal, by a flow of inert gas entering at port 6 and exiting at restricted port 7. Contained in the probe is a quartz light guide 8 terminated by lens 9 in the probe and split above the probe to lead to a laser 10 and spectrometer 11.

In operation, a pulsed beam from laser 10 is transmitted through light guide 8 and focussed by lens 9 onto the surface of the steel 1 causing ablation, ionisation and excitation. The resultant excited radiation is transmitted by lens 9 and light guide 8 to spectrometer 11 comprising diffraction grating, primary and secondary slits and detectors. The detectors will be photodiode arrays and/or photomultipliers capable of operating in the spectral region 250—1000nm and will have the facility of providing time resolution (gating) to eliminate recording of the initial burst of background radiation and optimise line to background ratios. Electrical signals, from detectors, corresponding to the intensities of the desired element lines and background measurements will be converted to percentage concentration and displayed as such by electronics unit 12.

Fig. 2 shows an alternative form for probe 4. The probe 4 functions generally as described above in relation to the arrangement of Fig. 1.

However, in this case the laser rods, flash lamps and 'Q' switch 13 are mounted in the top of the probe 4. Laser unit 13 is connected to its power and coolant supplies by cable and pipes 14. Lens 9 focusses the laser on the surface of metal 1. A second lens 15 transmits the excited radiation via light guide 16 to the spectrometer.

Laser excitation of the material (in this case liquid metal) provides a spectral continuum of radiation in addition to the characteristic line or lines sought. To improve the signal (spectral line) to noise (spectral continuum) ratio at the spectrometer this is time gated so as to reduce the initial high intensity background peak. This feature is illustrated in the graph of Fig. 3 of received excitation radiation against time. The laser pulse period (20 nano seconds) is represented at 20 with the time gate shut. The gate is opened as shown at 21 after approximately 9 micro seconds after initiation of the laser pulse. The received continuum radiation after attenuation is shown at 22, and the received carbon 909.4nm characteristic radiation at 23 with the time gate open.

The importance of the gate can be seen from the fact that without its use the peak of 22 would be larger by am approximate ratio of 1000:1, swamping the carbon characteristic peak.

For scrap (raw material) sorting the invention may be applied in a manner not illustrated by presenting individual pieces of scrap (raw material) over a small aperture by a continuous feed system. The laser beam is directed upwards at the specimen which is held stationary for such time as is necessary to ablate any non-representative material from the surface and carry out an analysis. A lens system is used to focus the laser excited emissions onto a light guide linked to a spectrometer. Monitoring of the various element emissions is used to determine the length of the stationary period and ensure a representative analysis is obtained. Time gating of the detectors is used to maximise line to background ratios for all elements measured. On conclusion of the analysis the material is automatically directed to the appropriate collection point.

CLAIMS

1. A method of analysing a material comprising the steps of exciting a portion of the material by means of a pulsed laser beam, transmitting the radiation produced by the excitation of the material through a light guide and analysing the radiation so transmitted in a spectrometer including facility for analysing in the infra-red region of the spectrum.

2. A method according to claim 1 wherein the spectrometer is gated to receive transmitted radiation a predetermined time after excitation of the material.

3. A method according to claim 1 or 2 wherein the material is liquid metal within a metallurgical vessel.

4. A method according to claim 3 wherein the liquid metal is steel.

5. A method according to claim 2 or 3 wherein the method is carried out in a continuous or semi-continuous sequential operation during a metallurgical process.

6. A method according to claim 1 or 2 wherein the meterial is a solid metal body.

7. A method according to any one of the preceding claims wherein the laser is subject in operation to variable tuning.

8. A method according to any one of the preceding claims wherein the laser is at least partially transmitted to the metal body by a light guide.

9. A method for analysing a metal body substantially as hereinbefore described with reference to the accompanying drawings.

10. Apparatus for analysing a material comprising means for generating a pulsed laser beam for exciting a portion of the material, a light guide for transmission of the radiation produced by the excitation of the material to a spectrometer; the spectrometer incorporating analysing means working in the infra-red region of the spectrum.

11. Apparatus according to claim 10

wherein the spectrometer includes gating means for receiving transmitted radiation a predetermined time after excitation of the material.

12. Apparatus according to claim 10 or 11 for analysing molten metal within a metallurgical vessel light guide is provided with thermal protection adjacent the metal body.

13. Apparatus according to clam 10, 11 or 12 including temperature sensing means arranged to operate using radiation from the metal body transmitted through the light guide.

14. Apparatus according to claim 10, 11, 12 or 13 wherein the laser is arranged to be transmitted at least partially to the metal body by a light guide.

15. Apparatus for analysing a metal body substantially as shown in and as hereinbefore described with reference to the accompanying drawings.

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